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DOI: https://doi.org/10.1007/s10266-018-0398-6

Originally published at:
Körner, Philipp; Sulejmani, Aljmedina; Wiedemeier, Daniel B; Attin, Thomas; Tauböck, Tobias T (2019). Demineralized enamel reduces margin integrity of self-etch, but not of etch-and-rinse bonded composite restorations. Odontology / the Society of the Nippon Dental University, 107(3):308-315.
DOI: https://doi.org/10.1007/s10266-018-0398-6

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Keywords: margin integrity, demineralized enamel, etch-and-rinse, self-etch adhesive, resin composite

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Abstract

The aim of this study was to investigate margin integrity of Class V composite restorations in demineralized and sound enamel after bonding with different etch-and-rinse and self-etch adhesive systems. Out of a total of 60 specimens from bovine incisors, 30 specimens were demineralized (21 days, acid buffer, pH 4.95) to create artificial enamel lesions. Circular Class V cavities were prepared in all 60 specimens and treated with either an unfilled etch-and-rinse adhesive (Syntac Classic; Ivoclar Vivadent), a filled etch-and-rinse adhesive (Optibond FL; Kerr), or a self-etch adhesive (iBond Self Etch; Heraeus) (n = 10 per group). The cavities were restored with a nanofilled resin composite and thermocycled (5000x, 5-55 °C). Scanning electron microscopy was used to evaluate margin integrity of the composite restorations, and the percentage of continuous margin was statistically analyzed (α = 0.05). Demineralized enamel led to significantly lower margin integrity when the self-etch adhesive iBond Self Etch was applied, but did not affect margin integrity when the etch-and-rinse adhesives Optibond FL (filled) or Syntac Classic (unfilled) were used. No significant differences in margin integrity in sound and demineralized enamel were observed between the different adhesives. Demineralized enamel reduces margin integrity of composite restorations when bonded with a self-etch adhesive, but does not affect margin integrity when an etch-and-rinse approach is used.

Introduction

Resin-based composites are increasingly popular for direct dental restorations [1,2]. These materials allow for lesion-oriented, minimally invasive caries treatment, since they can be adhesively bonded to the remaining tooth structure without the need for macro-mechanical retention [3].

However, dentists are often confronted with demineralized enamel margins during the process of caries excavation. Despite minimally invasive treatment approaches, caries excavation in daily routine commonly does not only include softened and infected dentin, but also demineralized enamel, and is often extended to sound enamel margins [4,5]. Especially in extensive areas of demineralized enamel margins, this concept may lead to a high and potentially disproportional loss of dental hard tissue [6]. Therefore, the question arises, whether minimally invasive caries excavation needs to be extended to sound enamel margins in order to obtain optimum margin integrity of the restorations, and thus to guarantee an optimum sealing ability.

Adhesive bonding materials differ in filler content, polymerization shrinkage and viscosity, which might influence their penetration in sound and demineralized enamel [7,8]. Due to its good performance in multiple laboratory studies and clinical research, the filled adhesive Optibond FL can be regarded as an established and well investigated etch-and-rinse adhesive [9–11]. The unfilled adhesive Syntac Classic and self-etch adhesive iBond Self Etch have also been used in various *in vitro* and *in vivo* studies and are considered to be sound representatives of their respective group [12–14]. Thus, the applicability of these adhesive systems and their influence on margin integrity of composite restorations in demineralized and sound enamel seem to be of great interest.

Therefore, the aim of the present study was to evaluate margin integrity of composite restorations in demineralized and sound enamel after application of these different etch-and-rinse and self-etch adhesive systems. The null hypotheses were that 1) margins in sound or demineralized enamel and 2) different kinds of adhesive systems do not affect margin integrity of composite restorations.

Materials and methods

Specimen preparation and demineralization

Fig. 1 illustrates the experimental design. A total of 60 specimens were prepared from the crowns of freshly extracted, undamaged, permanent bovine incisors stored in tap water until use, and randomly assigned into six groups of ten specimens each. The cementum layer was entirely removed by using polishing discs (Sof-Lex Pop-on; 3M ESPE, St. Paul, MN, USA). Enamel in groups 1-3 was demineralized by storing the specimens for 21 days at 37 °C in an acid buffer containing 3 mM $CaCl_2 \times 2\ H_2O$, 3 mM KH_2PO_4, 6 µM MHDP, 50 mM CH_3COOH, KOH (to adjust the pH to 4,95), and distilled water [15]. In order to keep the pH constant, the solution was renewed every day. The pulp cavum of the specimens was blocked and sealed with nail polish before demineralization to avoid internal demineralization. A cross-section of an artificially demineralized enamel lesion, recorded using a transmitted light microscope with a 10x objective (BX60; Olympus, Volketswil, Switzerland) and a CMOS color camera (DP74; Olympus), is shown in Fig. 2. The mineral density of demineralized and sound enamel was exemplarily measured by transverse microradiography (TMR) [16] on one additional specimen, and is given in Fig. 3. After demineralization of groups 1-3, standardized Class V cavities (diameter: 3 mm, depth: 2 mm, bevel edge: 1 mm) were prepared in the labial surfaces of all 60 specimens using spherical headed diamond burs (D126; Garant, Munich, Germany). The entire margin of the cavity was localized in enamel.

Bonding procedure

Cavities of the 60 prepared specimens were treated with either an unfilled etch-and-rinse adhesive (Syntac Classic; Ivoclar Vivadent, Schaan, Liechtenstein), a filled etch-and-rinse adhesive (Optibond FL; Kerr, Orange, CA, USA), or a self-etch adhesive (iBond Self Etch; Heraeus, Hanau, Germany). Detailed information and composition of the adhesives are given in Table 1. The bonding procedure in the different groups strictly based on the manufacturers' instructions for use, and was performed as follows:

Unfilled etch-and-rinse adhesive (Syntac Classic) - Groups 1 and 4:

Enamel and dentin surfaces of demineralized (n = 10, group 1) and not demineralized (n = 10, group 4) specimens were etched with 37% phosphoric acid gel (Total Etch; Ivoclar Vivadent) for 15 s before rinsing with water for 30 s. After gently air drying, the adhesive system (Syntac Classic; Ivoclar Vivadent) consisting of Syntac Primer (15 s), Syntac Adhesive (10 s) and Heliobond (15 s) was applied and light cured (20 s).

For photoactivation, a polywave LED curing unit (Bluephase G2; Ivoclar Vivadent) was used in all groups (1-6) at an output irradiance of at least 1,100 mW/cm^2. Output irradiance of the curing unit was checked periodically during the experiment with a calibrated power meter (FieldMaxII-TO; Coherent, Santa Clara, CA, USA).

Filled etch-and-rinse adhesive (Optibond FL) - Groups 2 and 5:

Prior to application of the filled etch-and-rinse adhesive (Optibond FL; Kerr), enamel and dentin surfaces of demineralized (n = 10, group 2) and not demineralized (n = 10, group 5) specimens were etched with 37% phosphoric acid gel for 15 s, and rinsed for 30 s. After primer application for 15 s and gently air drying for 5 s, the adhesive was applied for 15 s, gently air-thinned, and light cured for 20 s.

Self-etch adhesive (iBond Self Etch) - Groups 3 and 6:

Enamel and dentin surfaces of demineralized (n = 10, group 3) and not demineralized (n = 10, group 6) specimens were conditioned by applying the self-etch adhesive (iBond Self Etch; Heraeus) for 20 s, followed by gently air drying (5 s) and light curing (20 s).

Composite application and thermocycling

After application of the different adhesive systems, all 60 pretreated Class V cavities were restored in one increment with a nanofilled composite material (Filtek Supreme XTE; 3M ESPE; shade A2B, LOT N535229), and light cured for 20 s. Surgical scalpel blades (No. 12D; Gebr. Martin, Tuttlingen, Germany) were used to remove excess before the restorations were finished and polished with silicon instruments (Brownie Mini-Points and Greenie Mini-Points; Shofu Dental Corporation, San Marcos, CA, USA) and polishing brushes (Occlubrush; Kerr). A microscope was used at 25x magnification (Stemi 2000; Zeiss, Oberkochen, Germany) during placement of the restorations and in order to check them. Subsequently, the specimens were artificially aged by thermocycling – 5000 times in water between 5 °C and 55 °C, dwell time of 20 s in each temperature bath, transfer time of 10 s (Willytec; Gräfelfing, Germany) [17].

Assessment of margin integrity

Negative copies of each restoration were taken with an A-polyvinylsiloxane material (President Light Body; Coltène, Altstätten, Switzerland) after thermocycling. The impressions were poured with epoxy resin (Epoxyharz L; R&G Faserverbundwerkstoffe, Waldenbuch, Germany) to receive positive replicas, and subsequently glued to aluminum carriers (Cementit Universal; Merz&Benteli, Niederwangen, Switzerland). The replicas were sputter-coated with gold (Sputter SCD 030; Balzers Union, Balzers, Liechtenstein) [18] and subjected to a quantitative margin analysis using scanning electron microscopy at 20 kV and 200x magnification (Vega TS5136XM; Tescan, Brno, Czech Republic). Margin qualities were classified as “continuous”, “non-continuous” or “not judgeable” by one trained and blinded examiner, as performed in previous studies [19,20]. Margin integrity between enamel and restoration was expressed in percentage of “continuous margin” in relation to the entire judgeable margin [8,21].

Statistical analysis

As part of descriptive statistics, means and standard deviation were computed. Normality assumptions were checked using Kolmogorov-Smirnov and Shapiro-Wilk tests. Two-way ANOVA with the two factors “demineralization“ (two levels) and “adhesive system“ (three levels) including interaction was then fitted to the margin integrity data. Subsequently, differences in the percentage of continuous margins between sound and demineralized enamel within each adhesive system were analyzed using post-hoc t-tests. The level of significance was set at 5%. All statistical analyses and plots were done with the statistical software R version 3.2.2 (The R Foundation for Statistical Computing, Vienna, Austria; www.R-project.org).

Results

The percentage of continuous margins (margin integrity) in demineralized and sound enamel after application of the different adhesive systems is presented in Fig. 4. While two-way ANOVA revealed no significance for the factor “adhesive system“, the factor “demineralization“ showed significant influence on margin integrity ($p = 0.010$). No significant interaction between the two factors was observed.

Demineralized enamel led to significantly lower margin integrity when the self-etch adhesive iBond ($p = 0.039$) was applied, but did not affect margin integrity when the etch-and-rinse adhesives Optibond FL (filled) or Syntac Classic (unfilled) were used. No significant differences in margin integrity in sound and demineralized enamel were observed between the different adhesives. Representative SEM micrographs of continuous and non-continuous margins are shown in Figs. 5 and 6.

Discussion

The results of this study indicate that demineralized enamel leads to significantly lower margin integrity of composite restorations compared to sound enamel, in case that the self-etch adhesive iBond Self Etch is used, but does not significantly affect margin integrity when the etch-and-rinse adhesives Optibond FL or Syntac Classic are applied. Thus, the first null hypothesis was partly rejected.

Bovine teeth are often used in studies evaluating margin integrity [20,22]. Due to a high degree of homogeneity and a similar chemical structure to human enamel [23], they are considered to be an appropriate alternative to human enamel [24]. Artificial enamel lesions were shown to have a histological structure similar to white-spot lesions and enamel caries, and were produced in accordance with previous *in vitro* studies investigating demineralized enamel [8,16,25–27]. The lesion depths (Fig. 2) were comparable to those described in these studies. However, natural enamel lesions can be deeper than artificial lesions, which might affect resin penetration under natural conditions [28].

In the present study, composite restorations bonded with the self-etch adhesive showed similarly high margin integrity in sound enamel as the etch-and-rinse adhesives. This finding is in agreement with other studies investigating margin integrity of self-etch adhesives in sound enamel [29,30]. Nevertheless, there are also studies describing significantly lower margin integrity in case that self-etch adhesives are used [12,31].

Beside correct application, margin integrity of composite fillings is influenced by the type of etching, filler content, resin viscosity and penetration ability of adhesive materials [32,33]. As the different adhesives in this study showed similarly high margin integrity in sound enamel, the question arises, to what extend demineralized enamel margins are responsible for the significantly lower margin integrity in case the self-etch adhesive was applied in demineralized enamel. An important step in order to enable deep resin penetration is to establish an adequate etching pattern [34]. Therefore, surface layers must be removed sufficiently [35]. Specimens were etched and treated in accordance with manufacturers' instructions meaning that they were etched with 37% phosphoric acid before application of the etch-and-rinse adhesives or acidic self-etch monomers. Acidic monomers in self-etch adhesives are less potent in terms of etching efficacy compared to conventional acids, conceivably leading to an irregular etching pattern [26,36], and reduced resin penetration into the lesion bulk [37]. The present study suggests that the etching efficacy of the self-etch adhesive might be even lower in demineralized enamel which might have resulted in an insufficient removal of the demineralized surface layer combined with an irregular etching-pattern, and thus limited penetration.

Enamel margin integrity of self-etch adhesives has been shown to benefit from pre-etching with phosphoric acid [38,39]. Therefore, it would be of interest to investigate the influence of pre-etching demineralized enamel with phosphoric acid on margin integrity in further studies. A recent study showed that a positive effect on margin integrity of composite restorations placed in demineralized enamel can be achieved through infiltration of demineralized enamel with a caries infiltrant before application of a self-etch adhesive [8].

The good performance of Optibond FL and Syntac Classic (etch-and-rinse adhesives) in this study is in agreement with literature findings, where these adhesives consistently showed predictable bonding ability in both laboratory and clinical trials [14,40–43]. However, the present study also revealed that the factor adhesive system, other than the factor demineralization, does not influence margin integrity of composite restorations. Therefore, the second null hypothesis could not be rejected.

Within the limitations of the present *in vitro* study, it can be concluded that demineralized enamel reduces margin integrity of composite restorations bonded with a self-etch adhesive. In contrast to the self-etch approach, the tested etch-and-rinse adhesives were able to establish similar degrees of margin integrity whether restoration margins were located in demineralized or sound enamel.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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Table 1 Composition of the adhesive systems used in the present study according to manufacturers` information

Adhesive system (manufacturer)	**Composition**	**LOT number**
Syntac Classic (Ivoclar Vivadent, Schaan, Liechtenstein)	Primer: Dimethacrylates, maleic acid, solvent, stabilizer Adhesive: Dimethacrylates, maleic acid, glutaraldehyde, water Heliobond: Bis-GMA, TEGDMA, stabilizers and catalysts	U43425 V01074 T15984
Optibond FL (Kerr, Orange, CA, USA)	Primer: HEMA, GPDM, ethanol, water, CQ, BHT, PAMA Adhesive: Bis-GMA, HEMA, GDM, CQ, ODMAB, barium aluminumborosilicate, Na_2SiF_6, fumed silicon dioxide, gamma-methacryloxypropyltrimethoxysilane	5554307 5543327
iBond Self Etch (Heraeus, Hanau, Germany)	UDMA, 4-META, glutaraldehyde, acetone, water, photo-initiators, stabilizers	010901

4-META: 4-methacryloyloxethyl trimellitate anhydride; BHT: Butylhydroxytoluen; Bis-GMA: Bisphenol-A-glycidyldimethacrylate; CQ: Camphorquinone; GDM: Glycerol dimethacrylate; GPDM: Glycerol phosphate dimethacrylate; HEMA: 2-hydroxylethyl methacrylate; ODMAB: 2-(Ethylhexyl)-4-(dimethylamino)benzoate; PAMA: Phthailic acid monomethacrylate; TEGDMA: Triethylene glycol dimethacrylate; UDMA: Urethane dimethacrylate

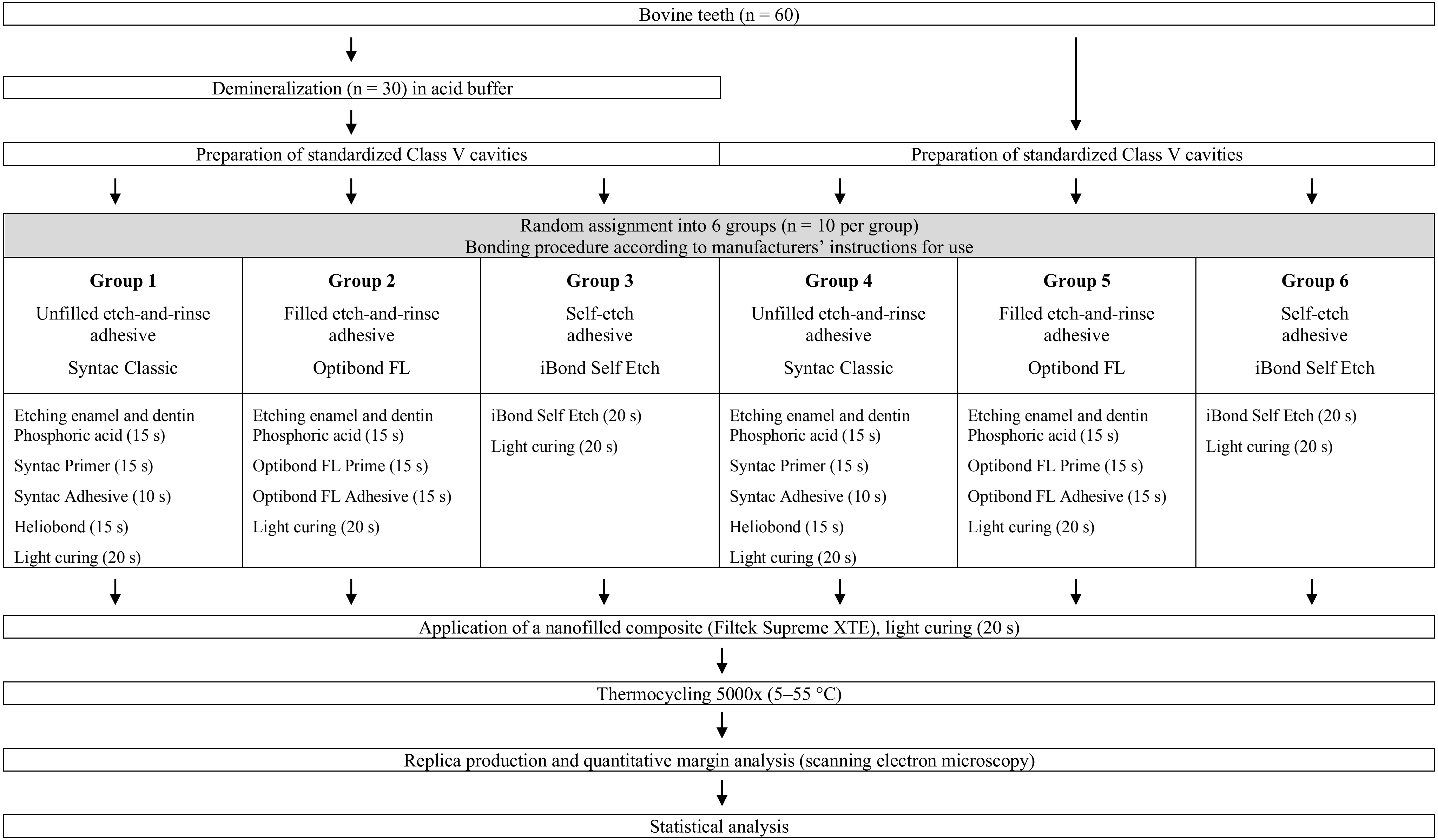

Fig. 1 Experimental design

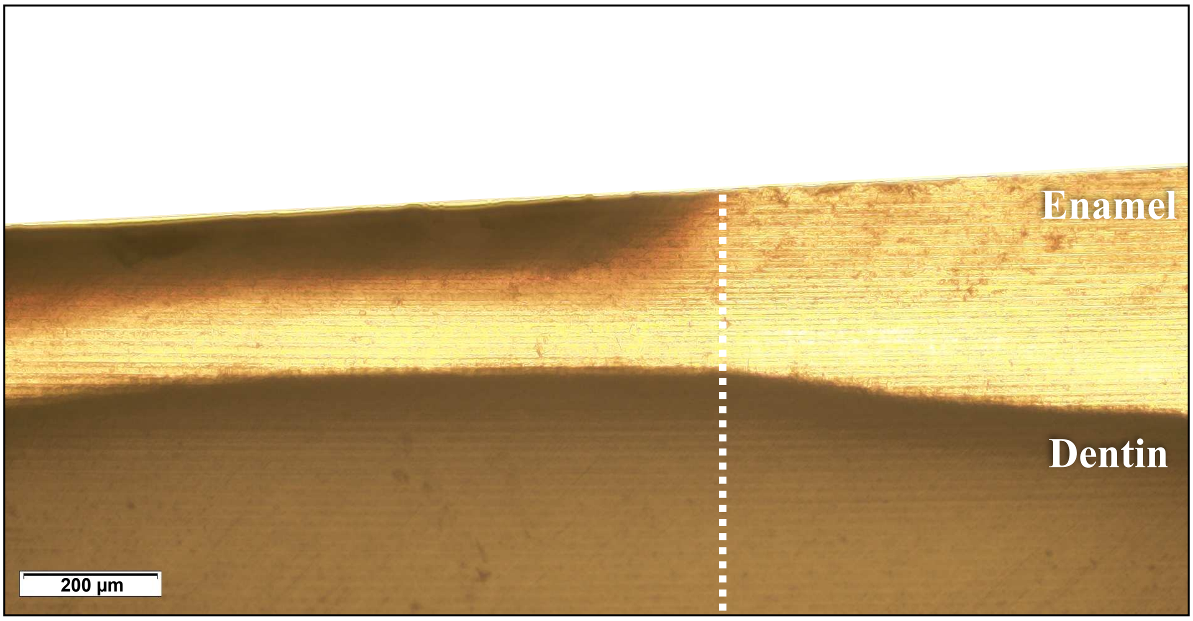

Fig. 2 Cross-sectional cut of an artificially demineralized enamel lesion after 21 days in acid buffer (left side) vs. no demineralization (right side)

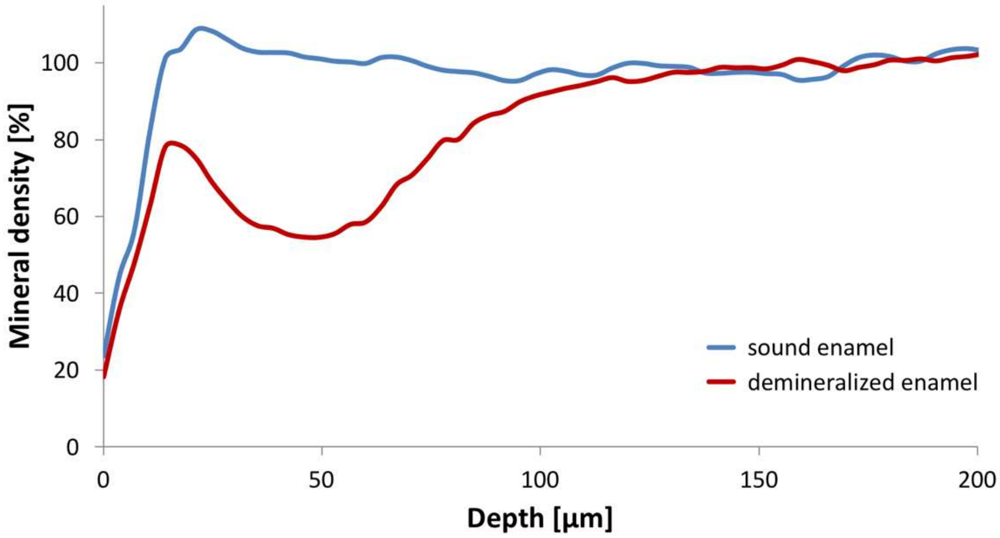

Fig. 3 Percentages of mineral density of sound and demineralized enamel related to the (lesion) depth in µm measured by transverse microradiography (TMR)

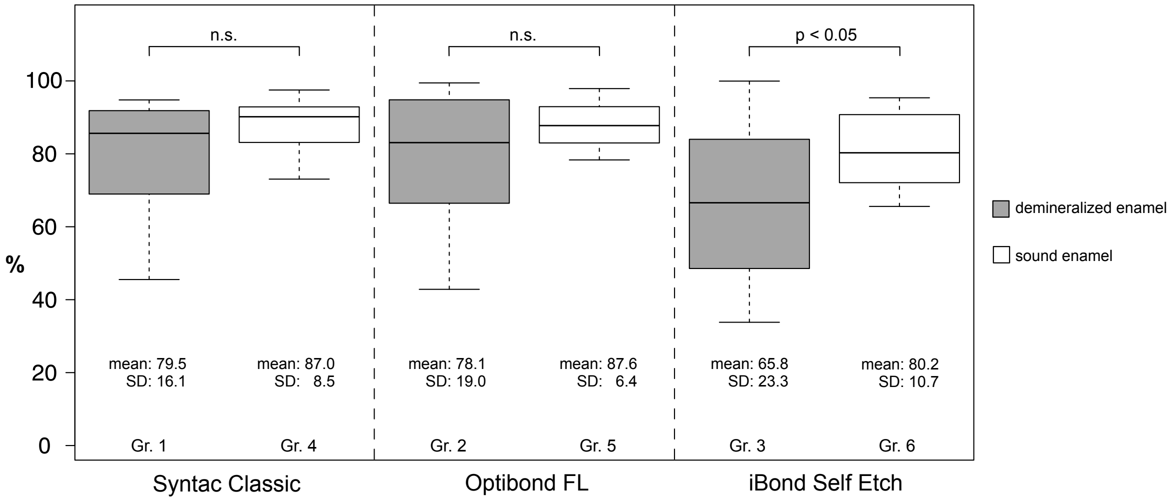

Fig. 4 Percentages of continuous enamel margins of composite restorations in demineralized and sound enamel, respectively, using Syntac Classic, Optibond FL, and iBond Self Etch as adhesives. The boxplots show the medians (black lines) with 25 and 75% quartiles (boxes); the whiskers represent 1.5*IQR (interquartile range) or minima and maxima of the distribution if below 1.5*IQR

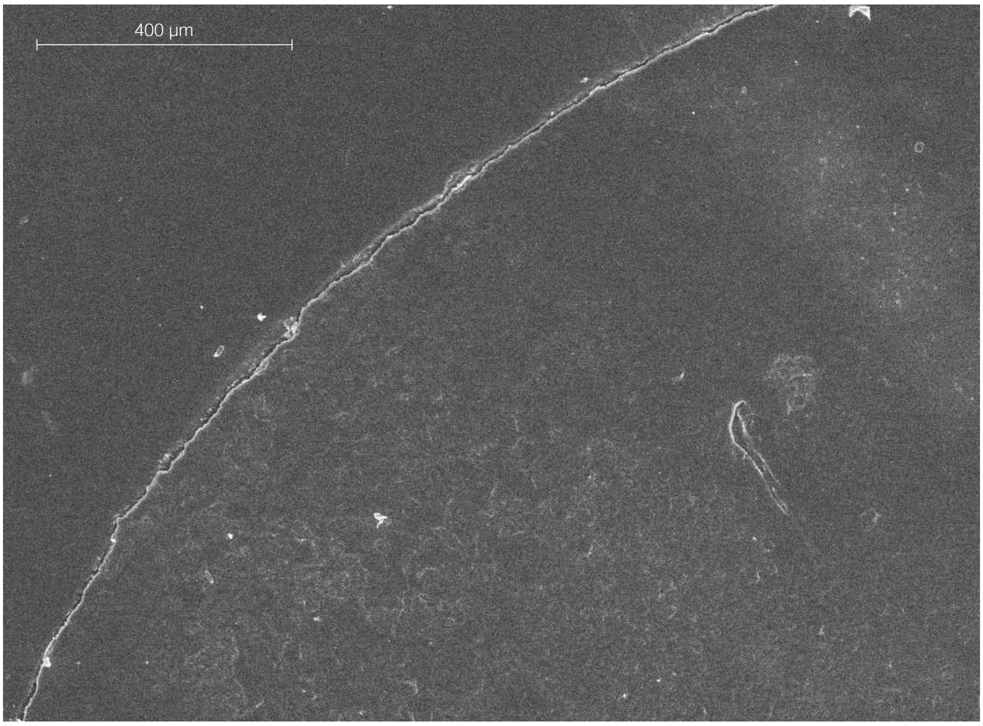

Fig. 5 SEM micrograph of non-continuous margin (group 3; iBond Self Etch in demineralized enamel)

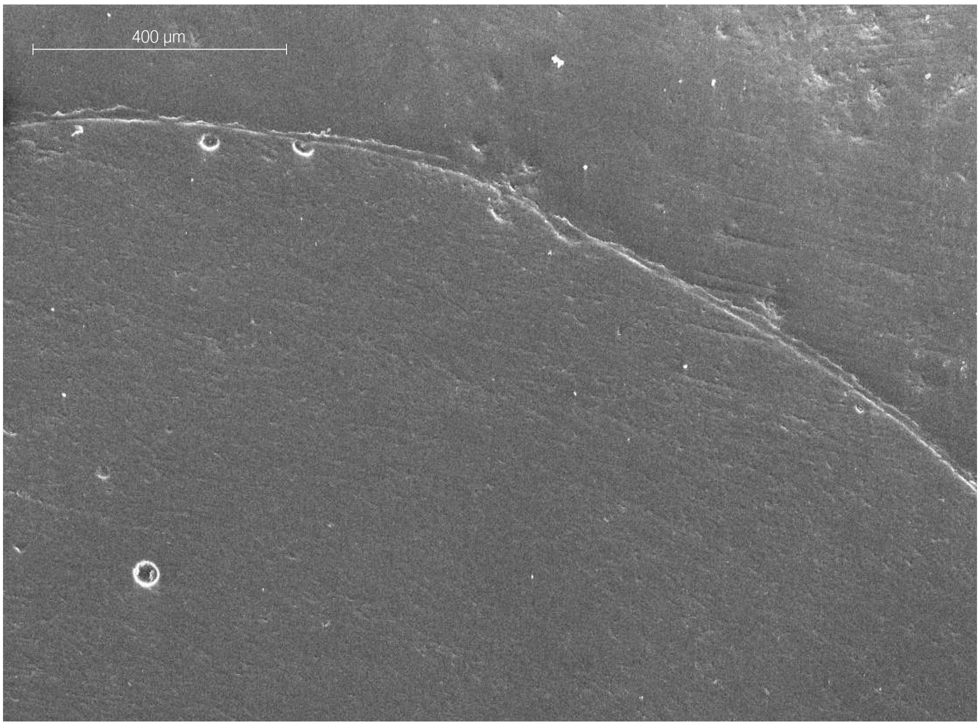

Fig. 6 SEM micrograph of continuous margin (group 6; iBond Self Etch in sound enamel)